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Separability Characteristics of Lubricating Greases

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Most devices employed to apply lubricating greases to bearings utilize pressure to control rate of flow. Their stability under pressure may therefore be of considerable practical importance. Separation of the mineral oil from greases, while in greasing appliances, may lead to concentration of the soap content to the point where the devices become clogged, and feed of new lubricant to the bearings is hindered or prevented. There is no method which is generally accepted for evaluating lubricating greases in this respect. Herschel [1] proposed a method which involved a press in which the grease sample was placed between discs of blotting and filter paper and the loss in weight of the sandwich was reported as the separation. Farrington and Humphreys[2] adopted that method for their study of the subject. The objective of this report is to outline another procedure and summarize the results obtained therewith. The construction of the tester is shown in Figure 1. The steps which preceded adoption of that design may be briefly summarized as follows:

1. Tests in series of three well-known commercial automatic cups showed that interlaboratory reproducibility was poor. The cups were not suitable for use in laboratory equipment because spring pressures varied appreciably and the pistons had a tendency to stick.

2. It was determined that even under favorable conditions, the tests using automatic cups were not only an evaluation of separability characteristics of the greases but were also an indication of variations of the clearances of the pistons.

3. It was found that pressure alone caused no important separation of the greases. Application of over 3000 pounds per square inch resulted in no appreciable separation of oil from greases which, when placed in automatic cups at pressures less than ten pounds per square inch, lost more than 25% of their mineral oil component during the same time interval and at the same temperature range.

4. It was concluded that separation in the cups was largely due to a filtering action and, therefore, the adoption of fritted glass plates in place of automatic cups was advisable because they are obtainable with a high degree of uniformity and may be accurately calibrated.

The test procedure developed is the following: The burette, the connecting tube and the funnel are filled with mineral oil so that the level of the oil in the burette is even with that of the filter plate. The oil used wherever possible is the same as that employed in the manufacture of the grease under test. Otherwise the oil is selected so that it has practically the same viscosity as that in the grease at the test temperature. After wiping any excess oil from the top of the filter plate, 150 grams of the grease are placed in the funnel and the yoke is made airtight by means of the connecting screws. The valves on the air reservoir, which maintain a constant pressure, are then opened. As the oil separates from the sample thru the filter plate, its volume is read directly from the burette. The tests are run in a constant temperature room or equivalent.

Using the fritted glass plates with pores of 5-10 microns, the extent of the agreement between the tester and the corresponding setup employing a series of automatic grease cups, is shown in the following tabulation:

(Continued on Page 3)

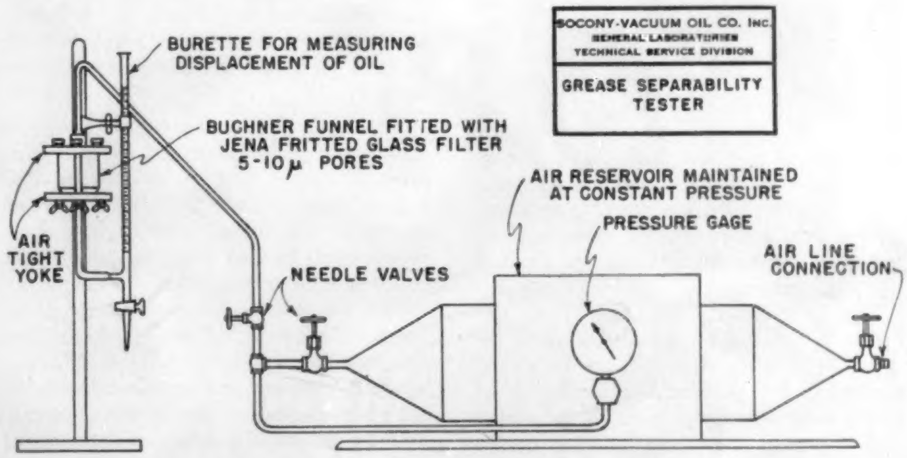

Fig. 1

[1] W. H. Herschel, Proc. Am. Soc. Testing Materials, 33 P. & I., 343-7 (1933).
[2] B. B. Farrington and R. L. Humphreys, Ind. Eng. Chem., 31, 230-5 (1939).

Improved Test Methods for Evaluation of Anti-Friction Bearing Greases Developed by Joint Committee of Annular Bearing Engineers Committee and National Lubricating Grease Institute

It is not unusual for bearing manufacturers to receive complaints on unsatisfactory performance which, upon investigation, are found actually to be due to some deficiency of the lubricant. Likewise, grease manufacturers sometimes receive complaints of unsatisfactory lubricant performance which are found actually to be due to failure of the customer to follow recommendations of the bearing manufacturer regarding operation and maintenance of the bearings.

The bearing and lubricating grease industries, recognizing the interrelated responsibilities for bearing performance, and acting through the Annular Bearing Engineers Committee and the Technical Committee of the National Lubricating Grease Institute, established in 1942 a Cooperative Committee on Test Methods for Anti-Friction Bearing Greases.

Although various test equipment and methods have been devised to compare and evaluate the properties and performance characteristics of anti-friction bearing greases, few of these have been generally accepted or adequately standardized. Moreover, the Armed Services faced with the necessity of procuring lubricating greases for widely varied purposes, have had to specify numerous special tests and test methods in Government Specifications which do not yield reproducible results in different laboratories, due to the fact time did not permit thorough study and standardization prior to issuance. Hence, the work of the Joint A.B.E.C.-N.L.G.I. Committee in developing and standardizing test methods for evaluation of anti-friction bearing greases has a direct relation to the war effort to an extent that is best appreciated by those working to supply the Armed Services with the best possible lubricants.

The Joint A.B.E.C.-N.L.G.I. Committee has to date issued three bulletins which describe in detail certain test methods and equipment for performance evaluation of anti-friction bearing greases. It is considered important that these bulletins receive wide distribution so that the methods described can be tried, checked, and compared by as many interested laboratories as possible.

The three bulletins recently issued are as follows:

TECHNICAL BULLETIN No. 1

Part 1—
Tentative Method for Determination of Low Temperature Torque Characteristics of Greases in Anti-Friction Bearings.

Part 2—
The S.O.D. Pressure-Viscosimeter.

Part 3—
The S.O.D. Pressure - Viscosimeter for Low Temperatures.

Part 4—
The Measurement of Low Temperatures by Thermal Electric Procedures.

TECHNICAL BULLETIN No. 4

Revised Norma - Hoffman Oxidation Test for Greases.

TECHNICAL BULLETIN No. 5

Tentative Method for Determination of Performance Characteristics of Lubricating Grease in Anti-Friction Bearings at Elevated Temperatures.

Copies of the above bulletins may be obtained through Annular Bearing Engineers Committee, 60 East 42nd Street, New York City, or from National Lubricating Grease Institute, Technical Committee, 164 Chandler Street, Buffalo, N. Y. A limited number of Bulletins 1 and 5 are available at $1.00 per copy and Bulletin 4 at $.50 per copy.

The need for greases which will give satisfactory performance and freedom from excessive viscous drag in anti-friction bearings of high altitude aircraft, where temperatures well below −60°F. have been encountered, focused attention on the lack of recognized methods for evaluating extreme low temperature characteristics of greases. Technical Bulletin No. 1, covering the work of the Joint A.B.E.C.-N.L.G.I. Committee on this problem, describes in detail a low temperature torque test apparatus. This equipment is designed to evaluate the torque characteristics of greases in anti-friction bearings at low rotational speeds and at extremely low temperatures.

Technical Bulletin No. 1 also contains detailed information on the S.O.D. Pressure-Viscosimeter which is used to determine grease viscosities over a range of temperatures and rates of shear. This information is included in the Bulletin to encourage further work on grease viscosity measurement, because potentially such work may yield data of a more fundamental nature than is obtainable by the use of torque test devices.

Bulletin 1 also describes recommended practices for accurate measurement of low temperatures. Measurement of extremely low temperatures involves technical difficulties not normally encountered. Since an error in temperature measurement of only a few degrees may involve very large errors in test results, a precise technique is essential.

Technical Bulletin No. 4 describes an improved procedure for the Norma-Hoffman Bomb Oxidation Test. While this test method has been in use for some years, it was never adequately standardized as to details of operation and procedure with the result that reproducibility between different laboratories has been poor. The revised method proposed in Bulletin 4 is a summation of the experiences gained from an extensive study by the Joint Committee, and work to date indicates it is more accurate, precise and reproducible. This test is designed to provide information which will permit predicting the behavior of greases in bearings in storage or in bearings contained in equipment, electric motors, instruments, etc., under storage conditions.

Technical Bulletin No. 5 describes a new test apparatus and procedure for evaluation of anti-friction bearing greases for high temperature service conditions. Aircraft accessories and equipment, in particular, contain bearings which operate at speeds as high as 10,000 RPM and at temperatures as high as 300°F. Since much of such equipment can only be re-lubricated at infrequent intervals, greases of exceptional properties are essential. Bulletin 5 proposes a test apparatus and operating procedure which is intended to provide information on the performance characteristics of greases under such conditions of high speed and high temperatures. It represents the first attempt to establish a method which can be used by all interested laboratories on a common basis.

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New Publications

We wish to announce a new publication by Petroleum Educational Institute, 704 South Spring Street, Los Angeles, Calif., entitled—DRIVING DIESELS.

This book presents in a non-technical manner the basic fundamentals of automotive diesels and will be of interest to drivers of diesel equipment.

Separability Characteristics of Lubricating Greases

(Continued from Page 1)

Table I

Automatic Grease Cup vs. Separability Tester 7.5 Lbs./Sq. In. Pressure — 70°F. Temperature

Conventional Cup Greases	*Total Hours Tested*	*Per Cent Separated Oil Cup*	*Tester*
Sample #1	168	9.1	8.2
Sample #2	168	6.6	7.1
Sample #3	72	3.7	4.1
Sample #3	168	4.9	5.6
Sample #4	168	4.6	4.3

The reproducibility of runs made in the Tester is shown by the data given in Table II:

Table II

#3 Cup Grease

Reproducibility of Runs in the Separability Tester

	Hours To Separate 5% Oil	*10% Oil*	*15% Oil*
Run 1	52	188	337
Run 2	57	197	355
Run 3	58	200	370

The variation found in the three runs is believed to be well within the range having practical significance.

Table III

Effect of Type of Soap in Oil Loss

No.	*Type*	*Grease Structure*	*ASTM Worked Penetration*	*Hours to Separate 5% Oil*	*10% Oil*	*25% Oil*
		Mineral Oil Component—S. U. Visc. @ 100°F.—105"				
1	Lithium	Buttery	255	3	8	21
2	Sodium	Tough-Fibrous	296	4	11	41
3	Calcium	Buttery	305	10	34	184
4	Aluminum	Smooth Gel	313.	17	62	319
		Mineral Oil Component—S. U. Visc. @ 100°F.—285"				
5	Sodium	Short Fibre	308	6	16	60
6	Calcium	Buttery	315	22	69	288
7	Calcium	Short Fibre	335	97	420	+1000

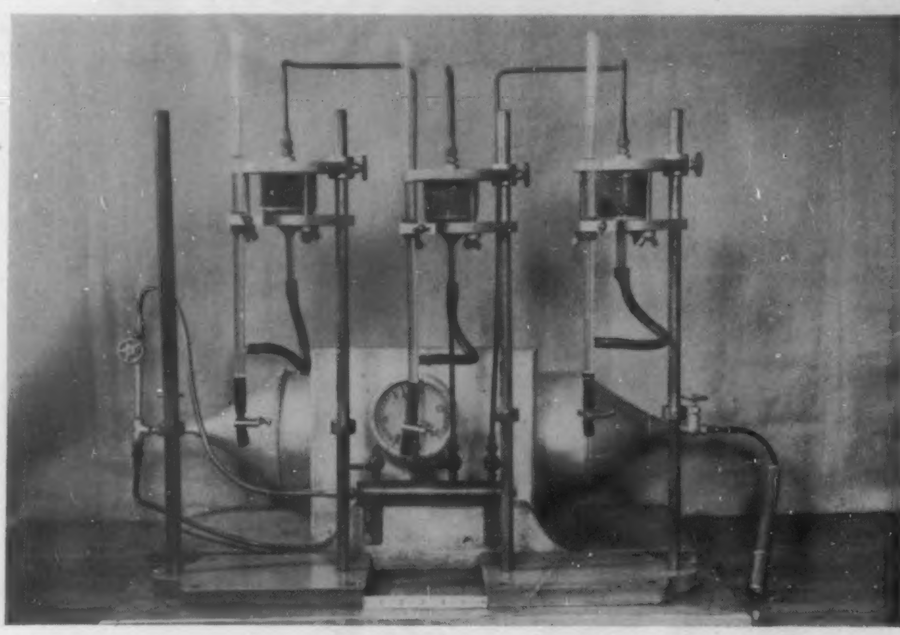
Experience has demonstrated that altho the above test procedure was primarily intended to rate the products in respect to their comparative pressure stabilities in automatic cups, the data also reliably indicate the relative tendencies of the greases to separate oil when subjected to pressures in centralized greasing systems and high pressure guns. A wide variation of greases have been tested and the data show that separability is determined by a number of factors all of which are of the type which affect the rate at which the oil may be filtered from the grease.

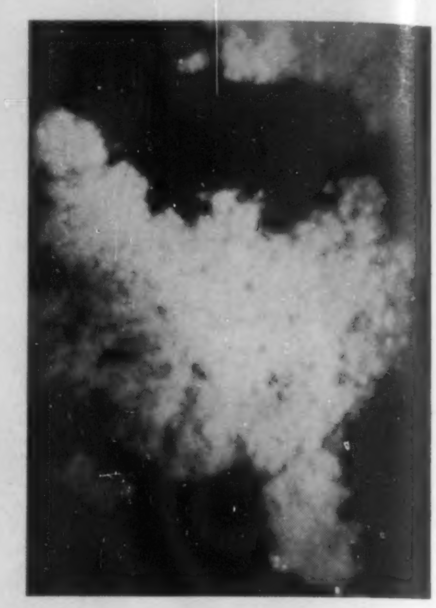
1. Structure
2. Percentage of Soap
3. Viscosity of Mineral Oil
4. Time
5. Pressure
6. Design of retaining agency.

The structures of greases are determined primarily by the type of soap, by the presence of additives as for example petrolatum and so-called coupling agents or mutual solvents and by manufacturing procedure. The type of mineral oil may also be a factor in some instances.

The first six products listed in Table III are conventional greases containing no additives. The two sodium base greases differ in the type of fatty material employed for making the soap. The short fibred calcium base product is not conventional in that it is not an emulsion of water-in-oil and is made with a novel combination of calcium soaps and a coupling agent or mutual solvent. The wide variation in structure of the soaps present in the seven samples may be illustrated by photomicrographs (Figures 2 thru 5). Grease No. 4 has the typical gel structure of aluminum base products while Greases Nos. 3 and 6 are water-in-oil emulsions normal for calcium base cup greases. When greases having conventional formulae are compared, Table III indicate that with other factors equal, such as percentage of soap and viscosity of mineral oil, lithium base products tend to be the least stable and aluminum base greases the most stable in respect to separation under the subject test conditions.

The consistency of greases is normally controlled by varying the percentage of soap. However, there are products the ASTM penetrations of which are the result more of the degree of working which they receive during manufacture than control of their soap contents. The effect of variations in concentrations of soap on separability may be illustrated by data (Table IV) obtained from tests of a line of conventional lime base cup greases made with a 105" at 100°F. viscosity pale oil.





GREASE No. 1
Dense mass, no fibrous structure evident.

GREASE No. 7
Fine uniform mass of hairlike appearing fibers.

TABLE IV

Effect of Percentage of Soap

Calcium Soap	ASTM Worked Penetration	Hours to Separate 5% Oil	10% Oil	25% Oil
10.6%	357	6	18	100
11.5%	305	10	34	184
16.8%	254	13	47	272
20.2%	200	47	185	+400

The soap is normally the component most responsible for the consistency of the grease and it is reasonable to expect that the higher the percentage of soap, the more difficult will it become to filter oil therefrom, particularly in large percentages.

For a given type soap base and procedure of manufacture, the lighter the viscosity of the mineral oil component, the greater will be the rate of separation of oil. This may be illustrated by referring to Table V showing the results of tests of a series of conventional cup greases made with the same calcium soap base.

TABLE V

Effect of Viscosity of Mineral Oil Component

S.U. Visc. @ 100°F.	% Soap	ASTM Worked Penetration	Hrs. to Separate 5% Oil	10° Oil
850″	15.0	255	95	245
500″	16.5	257	40	135
285″	15.5	262	24	103
105″	16.8	254	13	47

It is a truism to state that the viscosity of mineral oil varies with temperature. The structure of the grease may also undergo important changes, particularly if the temperatures pass thru certain critical ranges. Table VI illustrates the effect which an increase in test temperature from 70°F. to 130°F. had on three particular products.

TABLE VI

Effect of Temperature

Sample	70°F. 1 Day	4 Days	7 Days	130°F. 1 Day	2 Days
#1	2.6	5.9	8.0	8.4	11.3
#2	1.5	3.4	4.5	3.0	5.0
#3	10.0*	—	—	11.5*	—

*After 8 Hrs. *After 7½ Hrs.

Sample #1 is a conventional lime base cup grease made with an oil having a S.U. Viscosity of 850 seconds at 100°F. The second sample is a novel type of lime base grease containing an oil with a viscosity of 285 seconds at 100°F. Sample #3 is a lithium base product made with a 75 second at 100°F. viscosity oil. It may be of interest to note the extent to which separation of oil changed the concentration of soap in the top and bottom portions of the samples as they were removed from the tester after the runs at 130°F.

Sample	% Saponified Fat Before Test	After Test Top	Bottom
#1	12.9	12.8	20.3
#2	10.3	10.0	11.0
#3	14.8	18.2	23.3

It is obvious that time is a factor determining the amounts of oil that will be separated from the greases in the tester. The following data were taken from runs which were continued for 1000 hours.

TABLE VII

Effect of Time

Sample	ASTM Worked Penetration	% Oil Separation 200 Hrs.	500 Hrs.	1000 Hrs.
A	304	8.3	13.8	19.2
B	260	6.6	10.6	15.0

Samples A and B are conventional lime base cup greases made with an oil having a S.U. Viscosity of 850 seconds at 100°F.

Grease No. 2

Long tough relatively coarse fibrous structure.

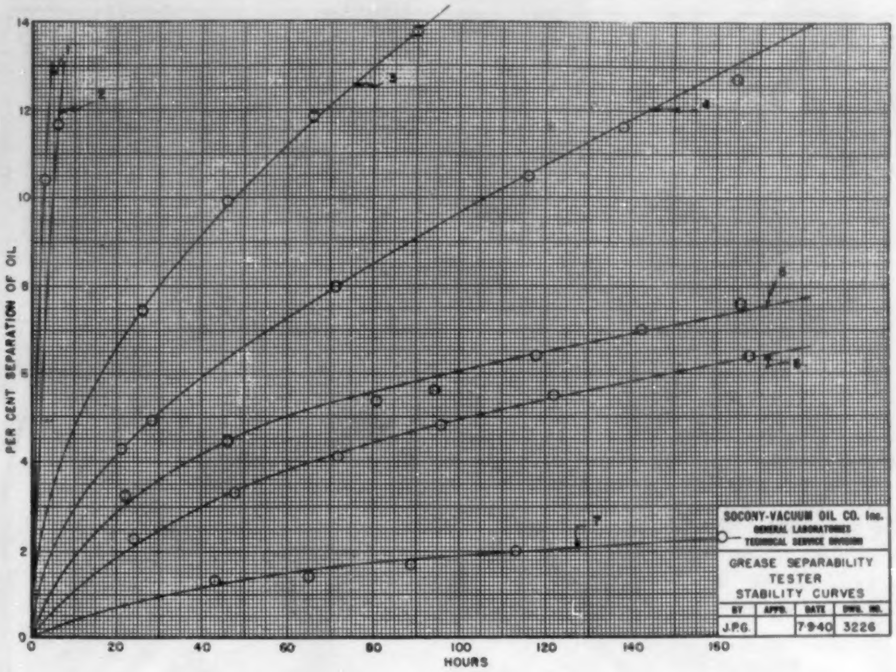
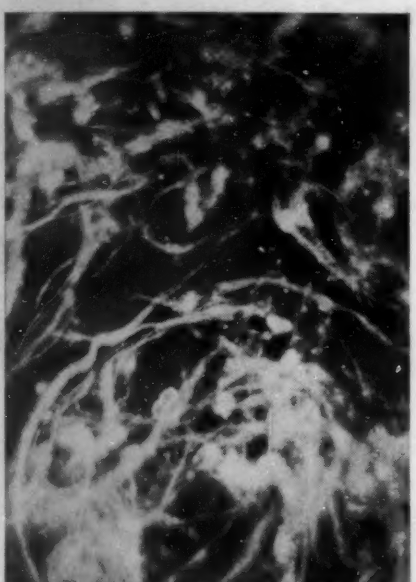


Fig. 6



Grease No. 5

Long definite fiber structure but not tough.

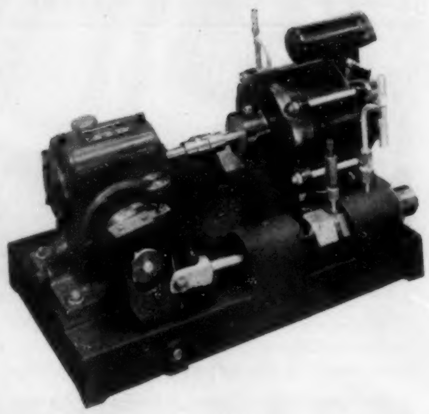
When the curves were plotted for those runs it was noted that their slopes still had developed no marked tendency to level or, in other words, the concentrating effect on the soap content of the grease in contact with the filter had still not led to important interference with rate of separation of the oil after 1000 hours. Figure #6 illustrates the form of curves obtained for greases of the type mentioned thruout this report. Curves Nos. 1 and 2 are soda base cup greases containing a 300 second oil. Nos. 3, 4, and 5 are conventional lime base greases made with oils having viscosities of 100, 285 and 850 seconds, respectively. No. 6 is a soda base grease with an oil heavier than 3000 seconds at 100°F., while #7 is a new type of lime base cup grease containing a 285 second at 100°F. oil.

Similar data could be presented to support the statements made earlier in this report that pressure is an important factor affecting the rate of separation. The design of the retaining agency as, for example, the effective clearance of the piston in the cup, is another variable the importance of which is self-evident.

Pressure is another important variable determining the amount of oil separation under the subject conditions but, as noted earlier in this report, its primary effect is that of promotion of the filtering action. When grease is completely confined, it has been observed that no separation occurs but when the confining agency is so designed that it may function as a filter then oil will be removed from the product as pressure is applied.

To summarize very briefly, separation of oil from grease while under pressure in application devices such as automatic cups, is essentially the outcome of a filtering action. In the rating of greases in terms of their separability characteristics under those conditions, it is advisable to recognize that there are a number of factors involved. Selection of the grease on the basis of type of soap base alone may not be sufficient to insure the desired stability.

TESTING ★ TOPICS



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